

DOCUMENT-IDENTIFIER: US 3033655 A

TITLE: Tube for detecting impurities in airTitle (1):Tube for detecting impurities in airOCR Scanned Text (3):

3 the individual chamber sections, these can be made of different colors. The construction according to the invention assures a satisfactory intimate contact between the air flowing through and the reagents. As has been said, the air resistance in the construction described is very low in the individual chambers. It is possible, however, if desired, to adjust the amount of air flowing through the different chambers according to the cross-section of the chambers. This can be done by changing the angles at which the longitudinal walls form with each other, so that the cross-section of the chambers and thus their volume is changed. In the form of the invention shown in FIGS. 3 and 4, the tube 8 is formed of glass, and is constructed at its ends in the same manner as in FIG. 1. At one point along its length, it is surrounded by a sleeve 9 of flexible plastic material. At this point, the tube is provided with a nick 10 to facilitate breakage. Inside the tube 8 is a separator 12, in the form of a cross, dividing the interior into a series of four chambers, in which are arranged four ampoules, 11a, 11b, 11c and 11d. As one example, the tube 11a may be used for testing for cyanogen chloride. The ampoule 11a is filled with a solution of 4-benzylpyridine and barbituric acid dissolved in acetone. This has no cover. Tube 11b is used for testing for phosgene. It contains a solution of N-phenylbenzylamine in alcohol. It has a cotton cloth jacket impregnated with sodium carbonate solution and dried. Tube 11c is for testing for Lewisite. The ampoule contains a solution of Michler's thio ketone in alcohol. There is no cloth cover. Tube 11d is used for testing for N-Lost (mustard gas). It contains a solution of Dragendorff's reagent (potassium bismuth iodide in ethyl acetate or ether). The outer covering of the ampoule is a filter paper impregnated with silica gel. Another possible arrangement is shown in FIG. 5, in which a T-shaped insert is used which divides the interior into two chambers of equal volume and a third chamber of double the volume of either of the first two chambers. It is also possible through varying the dimension of the inlets into the chambers to change the resistance to the air flow and thereby the throughput of air. For example if, in the form of FIG. 5 it is desired to use one of the reagents in twice the quantity of each of the other two, this can be placed in the largest chamber and the entrance to the chamber can be so dimensioned that the flow of air through all three chambers is practically equal. While I have described herein some embodiments of my invention, I wish it to be understood that I do not intend to limit myself thereby except within the scope of the claims hereto or hereinafter appended. I claim: 1. A device for testing gases comprising an elongated transparent tubular member, separating means within the member comprising at least one wall extending longitudinally within the tubular member and dividing it into compartments, glass ampoules containing reagents within said compartments, at least one of said glass ampoules having an outer covering of liquid-absorbent material, said tubular member having a portion deformable to permit breaking of the ampoules from the outside of the tube. 2. A device as claimed in claim 1 in which said separating means comprises an insert. 3. A device as claimed in claim 2 in which the tubular member and insert are of flexible material. 4. A device as claimed in claim 1 in which the tubular member is of flexible material. 5. A device as claimed in claim 1 in which the tubular member is of glass and the deformable portion is a breakable portion, and a sleeve of transparent flexible material around the breakable portion. 6. A device as claimed in claim 1 in which the liquid absorbent material comprises a sleeve on the outside of the ampoule. 7. A device as claimed in claim 6 in which the sleeve is formed of cotton cloth. 8. A device as claimed in claim 1 in which the liquid absorbent material is paper. References Cited in the file of this patent UNITED STATES PATENTS 1,333,850 Kennedy ----- Mar. 16, 1920 2,908,555 Grosskopf ----- Oct. 13, 1959 45

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File: PGPB

Oct 30, 2003

PGPUB-DOCUMENT-NUMBER: 20030203495

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DOCUMENT-IDENTIFIER: US 20030203495 A1

TITLE: Diagnostic test for elemental imbalances

PUBLICATION-DATE: October 30, 2003

## INVENTOR-INFORMATION:

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APPL-NO: 10/423130 [PALM]

DATE FILED: April 24, 2003

## RELATED-US-APPL-DATA:

Application is a non-provisional-of-provisional application 60/375566, filed April 25, 2002,

INT-CL-PUBLISHED: [07] G01N 31/22, G01N 33/20

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CIPS	G01 N 31/22	20060101
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REPRESENTATIVE-FIGURES: 2

## ABSTRACT:

A self-diagnostic test, a self-diagnostic test/apparatus, and method of manufacturing a self-diagnostic test for screening for elemental mineral imbalances in a patient utilizing an analysis of the reaction of mineral specific reagents to a sample from a patient are provided. In one embodiment, the invention is directed to a test for those elements that occur naturally in the body. In such an embodiment, the invention may test for those elements that comprise about 0.001% of the body weight or less (microtrace), those elements that comprise about 4% of the body weight or less (trace), those elements that comprise up to 96% of the body weight (mass), or any combination of the above.

## CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application is based on U.S. Application No. 60/375,566, filed on Apr.

.25, 2002, the disclosure of which is incorporated by reference.

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DOCUMENT-IDENTIFIER: US 20030203495 A1

TITLE: Diagnostic test for elemental imbalances

Abstract Paragraph:

A self-diagnostic test, a self-diagnostic test apparatus, and method of manufacturing a self-diagnostic test for screening for elemental mineral imbalances in a patient utilizing an analysis of the reaction of mineral specific reagents to a sample from a patient are provided. In one embodiment, the invention is directed to a test for those elements that occur naturally in the body. In such an embodiment, the invention may test for those elements that comprise about 0.001% of the body weight or less (microtrace), those elements that comprise about 4% of the body weight or less (trace), those elements that comprise up to 96% of the body weight (mass), or any combination of the above.

Detail Description Paragraph:

[0024] This present invention is directed to a self-diagnostic test kit for screening for elemental mineral imbalances in a patient, referred to as the diagnostic test herein.

Detail Description Paragraph:

[0048] In step 5, once the reagents are deposited and cured the web is finished and packaged for sale. Such a step may include many sub-steps including: cutting the web to a suitable size and shape; attaching the web to suitable product packaging; printing any instructional or consumer warning messages onto the packaging; and boxing or wrapping the test for final shipment. For example, the finishing process in one exemplary embodiment includes: applying a bead of adhesive to the back side of the web at prescribed locations; perforated rotary die cutting the web to a specified size and shape; attaching the perforated portion of the web to a sample container; and printing instructions and scale information on the web or box and boxing the test for shipment.

Detail Description Paragraph:

[0052] Finally, it should be understood that although this test is designed to provide a method of allowing self-diagnosis of an elemental imbalance by a user, the diagnostic test is not designed to provide quantitative information about the imbalance. It would be expected, and should be provided in the instructions of the test, that a user finding a positive indication of an elemental imbalance immediately contact a physician for a detailed quantitative analysis of the particular elemental imbalance found by the screening test of the current invention. Accordingly, it should be understood that while the test is designed for home use, it could also be utilized in a hospital setting as a screening test in combination with a more quantitative test available from a hospital laboratory. In one exemplary embodiment, the packaging of the home diagnostic test may include a list of the quantitative test codes or diagnostic codes suggested by the federal government, or a particular hospital or insurance provider given a particular diagnosis indication by the diagnostic test of the present invention.

## CLAIMS:

9. The self-diagnostic test as described in claim 5 wherein the mineral specific

reagents are selected from the group consisting of azomethine-H; chromotropic acid; dinitronaphthalenediol; 3,5-di-t-butylcatechol; 2,6-dihydroxybenzoic acid; curcumin; 5-Br-PAPS; o-nitrophenylfluorone; diphenylcarbazine; 5-Br-PADAP; BTAMB; TAMSMB; 5-Cl-PADAB; dithizone; 3,5-diBr-PAMB; nitroso-DMAP; nitroso-PSAP; nitroso-DEAP; 5-Br-PADAB; bathocuproin disulfonic acid disodium salt; bathocuproin; 3,5-diBr-PAESA; sodium bicinchoninate; neocuproin; 5-Br-PSAA; TMPyP; Na-DDTC; alufosone; chromazurol S; phenylfluorone; K.sub.2HgI.sub.4/I.sub.2; bindschedler's green leuco base; diphenylcarbazon: tris(1,10-phenanthroline)Fe(II) complex; bathophenanthroline disulfonic acid disodium salt; TPTZ; PDTs; PDT; nitro-PAPS; PPKO; ferrene S; PAR; oxine; DDTC; toluene-3,4-dithiol; PAN; dimethylglyoxime; bismuthiol-2; 2,3-diaminonaphthalene; PV; SATP; toluene-3,4-dithiol; henylfluorone 3,3-diaminobenzidine; o-phenylenediamine; 4-chloro-o-phenylenediamine; ammonium molybdate; malachite green; BPA; zincon; XO; TMPyP; zinquin ethyl ester; and T(5-St)P.

10. The self-diagnostic test as described in claim 6 wherein the mineral specific reagents are selected from the group consisting of PC; MX; indo 1; indo 1-AM; chlorophosphonazo-III; neo-thorin; fluo 3; fluo 3-AM; arsenazo-III; HDOPP-Ca; rhod 2; rhod 2-AM; GHA; quin 2; quin 2-AM; calmagite; fura 2; fura 2-AM; thio-michler's ketone; MQAE; SPQ; diethylcarbamate-Cu; diphenylcarbazon; triocytlin; tris(1,10-phenanthroline)Fe(II); Co(3)-5-Cl-PADAP; malachite green; bis(12-crown-4); nitrophenylazo-15-crown-5; oxine; pararosaniline; barium chloranilate; methylene blue; O-phthalaldehyde; p-phenylenediamine; tris[2-(phenyliminomethyl)pyridinato] iron; and 2-aminoperimidine HCl/HBr.

11. The self-diagnostic test as described in claim 7 wherein the mineral specific reagents are selected from the group consisting of lumogallion; o,o'-dihydroxyazobenzene; aluminon; oxine; 5-Br-PADAP; rhodamine B; brilliant green; arsemate; thionalide; nitrocatechol; ethyl violet; dimethylsulfonazo-III; sulfonazo-III; chlorophosphonazo-III; chromazural S; arsenazo-I; acetylacetone; beryllon-III; 2-methyloxine; bismuthio-II; XO; DDTC; dithizone; bindschedler's green leuco base; diphenylcarbazon; PAN; formaldoxime; pyrogallol red-AM; cesibor tetraphenylborate; EuAc.sub.3 Eu.sub.20.sub.3; GdAc.sub.3; Gd(NO.sub.3).sub.2; sincon; semiethylxylenol Blue; KAUCN.sub.2; NaAuCl.sub.4; KAUCl.sub.4; KAUI.sub.4; 5-(p-dimethylaminobenzylidene)rhodamine; PAR; K.sub.3IrCl.sub.6; Na.sub.3IrCl.sub.6; SnCl.sub.2-HBr; leuco-crystal violet; PbAc.sub.2; PbCl.sub.2; Pb(NO.sub.3).sub.2; MePbAc; TPPS; thorin; bibenzyl-14-crown-4; phosphododecyl-14-crown4; TTD-14-crown-4; methyl dodecyl-12-crown-4; dibenzothiazolylmethane; EtHgCl.sub.2; EtHgphosphate; Hg(CN).sub.2; EtHgthiosalicylate (thiomersal); mersalyl; PCMB; PHMB; PhHgAc; HgCl.sub.2; HgAc.sub.2; HgSO.sub.4; mercurochrome; Baker's reagent (2Hg); tetrakismercuroacetate (TAM) (4Hg); STTA; thio-Michler's ketone; di-alpha-naphthylthiocarbonate; sulfochlorophenol-S; TPAC; BPR; phenylfluorone; Os(NH.sub.3).sub.6I.sub.3-; K.sub.2OsCl.sub.6; K.sub.2OsO.sub.4; tiron; K.sub.2PdCl.sub.4; K.sub.2PdBr.sub.4; K.sub.2PdI.sub.4; PdCl.sub.2; Pd(NO.sub.3).sub.2; BTAMB; 5-Br-PSAA; 5-Br-PAPS; thiooxine; p-nitroso-N,N'-dimethylaniline; K.sub.2PtCl.sub.4; K.sub.2PtCl.sub.6; K2PtI.sub.6; K.sub.2Pt(NO.sub.2).sub.4; Pt(NH.sub.3).sub.2Cl.sub.2; Pt (ethylenediamine)Cl.sub.2; K.sub.2Pt(CN).sub.4; ReCl.sub.3; 2-furildioxime; dimethylglyoxime; methylene blue; kalibor; TPTZ; 1,10-phenanthroline; SmAc.sub.3; Sm(NO.sub.3).sub.3; SmCl.sub.4; 5,7-dichloro-oxine; quinizarin; AgNO.sub.3; KAgCN.sub.2; 3,5-diBr-PADAP; 3,5-diBr-PAESA; 2-amino-6-methylthio-4-pyrimidine-carboxylic acid; PC; dinitrosulfonazo-III; murexide; bismuthiol-2; diethyldithiocarbamate; malachite green; Th(NO.sub.3).sub.4; arsenazo-III; morin; diantipyrylmethane; 0,0'-dihydroxyazobenzene; crystal violet; alizarin; Na.sub.2WO4; toluene-3,4-dithiol; UO.sub.2Ac.sub.2; K.sub.3UO.sub.2F.sub.5; UO.sub.2(NO.sub.3).sub.2; UO.sub.2SO.sub.4; TbCl.sub.3; YbAc.sub.3; Zr (NO.sub.3).sub.4; PV; TAN; and alizarin red S.

14. A self-diagnostic test apparatus comprising: a body having at least one biological fluid receptacle disposed thereon; a biological sample conduit in fluid

communication with the at least one biological fluid receptacle; and a plurality of mineral specific reagents disposed such that each mineral specific reagent may be exposed to a biological sample deposited within the at least one biological fluid receptacle, the mineral specific reagents being selected to react with a different selected mineral within the biological sample such that when the selected mineral specific reagent is exposed to a sufficient concentration of the selected mineral in the biological sample a visible change is induced in the selected mineral specific reagent.

15. The self-diagnostic test apparatus as described in claim 14 comprising a plurality of biological fluid receptacles wherein each of the plurality of mineral specific reagents is independently disposed within a different one of the plurality of biological fluid receptacles.

16. The self-diagnostic test apparatus as described in claim 14 wherein each of the plurality of mineral specific reagents is disposed on a different at least one substrate removably disposed within the at least one biological fluid receptacle.

17. The self-diagnostic test apparatus as described in claim 16 wherein each of the different at least one substrates is a dipstick.

18. The self-diagnostic test apparatus as described in claim 14 wherein at least a portion of the body is transparent such that the visible change of the selected mineral specific reagents may be externally viewed.

19. The self-diagnostic test apparatus as described in claim 14 wherein the plurality of mineral specific reagents are selected to detect at least one mineral from a mineral family selected from the group consisting of microtrace, trace, mass, and all naturally occurring.

20. The self-diagnostic test apparatus as described in claim 14 wherein the plurality of mineral specific reagents are selected to detect at least one mineral that does not occur naturally within the human body.

21. The self-diagnostic test apparatus as described in claim 14 wherein the plurality of mineral specific reagents are selected to detect a mineral imbalance indicative of a disorder selected from the group consisting of ADD/ADHD, Alzheimer's disease, anemia, ataxia, bipolar disorder, birth defects, blood disorders, brain damage, brain disease, breast cancer, breathing disorders, bone cancer, cardiomyopathy, general cancer, Crohn's disease, depressive disorders, encephalopathy, eye damage, heart damage, high blood pressure, infertility, intestinal disorders, leishmaniasis, liver cancer, liver damage, lung damage, lung disease, lung cancer, kidney damage, kidney disease, manic disorders, nerve damage, neuropathy, organ damage, pancreatic cancer, periodontal disease, psychosis, renal failure, skin disorders, and Wilson's disease.

22. The self-diagnostic test apparatus as described in claim 14 wherein the plurality of mineral specific reagents are selected to detect an imbalance in a plurality of minerals selected from the group consisting of B, Ge, F, I, Si, V, Cr, Co, Cu, Fe, Ni, Mo, Se, Zn, Sn, and Mn.

23. The self-diagnostic test apparatus as described in claim 14 wherein the biological sample is selected from the group consisting blood, urine, saliva, mucous, tears, and hair.

24. The self-diagnostic test apparatus as described in claim 14 wherein the visual change is a calorimetric change.

DOCUMENT-IDENTIFIER: US 5174959 A

**\*\* See image for Certificate of Correction \*\***

TITLE: Breath component monitoring device

Brief Summary Text (10):

Fortune, U.S. Pat. No. 2,186,902 discloses the use of soluble nitroprusside chromogens in the presence of ammonia and soluble carbonates for the detection of what was termed "acetone" (actually acetoacetic acid) in urine samples. Varying colorations are observable for the quantitative determination of "acetone" levels.

DOCUMENT-IDENTIFIER: US 20030021744 A1

TITLE: Process for removing hydrogen sulfide from gas streams which include or are supplemented with sulfur dioxide, by scrubbing with a nonaqueous sorbent

Summary of Invention Paragraph:

[0004] The present inventor's U.S. Pat. No. 5,738,834, the entire disclosure of which is hereby incorporated by reference, discloses a process which uses a sulfur-amine nonaqueous sorbent (SANS) and operating conditions under which sulfur itself can convert hydrogen sulfide to polysulfides which are nonvolatile but which can be readily transformed to sulfur by reaction with an oxidizing agent. This is done in a solvent with a high solubility for sulfur so that solid sulfur formation does not occur in the absorber or in the air-sparged regenerator. Solid sulfur formation can be initiated in process equipment designed to handle solids and can be done under well-controlled conditions. In the SANS process, the sour gas is fed to an absorber (typically countercurrent) where the H.sub.2S is removed from the gas by a nonaqueous liquid sorbing liquor which comprises an organic solvent for elemental sulfur, dissolved elemental sulfur, an organic base which drives the reaction converting H.sub.2S sorbed by the liquor to a nonvolatile polysulfide which is soluble in the sorbing liquor, and an organic solubilizing agent which prevents the formation of polysulfide oil--which can tend to separate into a separate viscous liquid layer if allowed to form. The solubilizing agent is typically selected from the group consisting of aromatic alcohols and ethers including alkylaryl polyether alcohol, benzyl alcohol, phenethyl alcohol, 1-phenoxy-2-propanol, 2-phenoxyethanol, alkyl ethers including tri(propylene glycol) butyl ether, tri(propylene glycol) methyl ether, di(ethylene glycol) methyl ether, tri(ethylene glycol) dimethyl ether, benzhydrol, glycols such as tri(ethylene) glycol, and other polar organic compounds including sulfolane, propylene carbonate, and tributyl phosphate, and mixtures thereof. The sorbing liquor is preferably essentially water insoluble as this offers advantages where water may be condensed in the process. It is also preferable for water to be essentially insoluble in the solvent. The nonaqueous solvent is typically selected from the group consisting of alkyl-substituted naphthalenes, diaryl alkanes including phenylxyl ethanes such as phenyl-o-xylene, phenyl tolyl ethanes, phenyl naphthyl ethanes, phenyl aryl alkanes, dibenzyl ether, diphenyl ether, partially hydrogenated terphenyls, partially hydrogenated diphenyl ethanes, partially hydrogenated naphthalenes, and mixtures thereof. In order to obtain a measurable conversion of sulfur and hydrogen sulfide to polysulfides, the base added to the solvent must be sufficiently strong and have sufficient concentration to drive the reaction of sulfur and hydrogen sulfide to form polysulfides. Most tertiary amines are suitable bases for this use. More particularly, tertiary amines including N,N dimethyloctylamine, N,N dimethyldecylamine, N,N dimethyldodecylamine, N,N dimethyltetradecylamine, N,N dimethylhexadecylamine, N-methyldicyclohexylamine, tri-n-butylamine, tetrabutylhexamethylenediamine, N-ethylpiperidine hexyl ether, 1-piperidineethanol, N-methyldiethanolamine, 2-(dibutylamino)ethanol, and mixtures thereof are suitable for use in the said process. It should be noted that while the solvent utilized in the process requires the addition of a base to promote the reaction of sulfur and hydrogen sulfide to form polysulfides, the base and the solvent may be the same compound.





US 20030021744A1

(19) **United States**(12) **Patent Application Publication** (10) **Pub. No.: US 2003/0021744 A1**  
DeBerry et al. (43) **Pub. Date: Jan. 30, 2003**(54) **PROCESS FOR REMOVING HYDROGEN  
SULFIDE FROM GAS STREAMS WHICH  
INCLUDE OR ARE SUPPLEMENTED WITH  
SULFUR DIOXIDE, BY SCRUBBING WITH A  
NONAQUEOUS SORBENT****Publication Classification**(51) **Int. Cl.<sup>7</sup>** ..... **B01D 53/52**  
(52) **U.S. Cl.** ..... **423/228; 423/576.4**(76) **Inventors: David W. DeBerry, Austin, TX (US);  
Dennis Dalrymple, Georgetown, TX  
(US); Kevin S. Fisher, Georgetown,  
TX (US)**(57) **ABSTRACT****Correspondence Address:  
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The invention relates to improvements in a known process and system wherein hydrogen sulfide is removed from a gaseous stream, using a nonaqueous scrubbing liquor in which are dissolved sulfur and a reaction-promoting amine base. In a first aspect of the invention sulfur dioxide is added to the sulfur-amine nonaqueous sorbent (or advantage is taken of SO<sub>2</sub> which may already be present in the gas stream) to obtain better H<sub>2</sub>S removal, lower chemical degradation rates, and lower rates of formation of byproduct sulfur salts. In a further aspect of the invention the gas to be treated is mixed with oxygen and passed through an oxidation catalyst reactor to either effect oxidation of part of the H<sub>2</sub>S to form the required amount SO<sub>2</sub> for reaction with the remaining H<sub>2</sub>S, or to effect partial oxidation of the H<sub>2</sub>S in the feed gas to form elemental sulfur, or to form various combinations of products as desired for the application, prior to scrubbing with the nonaqueous solvent.

(21) **Appl. No.: 10/190,448**(22) **Filed: Jul. 5, 2002****Related U.S. Application Data**(63) **Continuation-in-part of application No. 09/503,898,  
filed on Feb. 15, 2000, now Pat. No. 6,416,729.**(60) **Provisional application No. 60/156,545, filed on Sep.  
29, 1999.**

DOCUMENT-IDENTIFIER: US 6544492 B1

TITLE: Regeneration method for process which removes hydrogen sulfide from gas streams

Detailed Description Text (3):

Absorber 11 is a conventional liquid-gas contact apparatus at which the input gas stream 22 to be purified is passed in counter-current or other relation to a liquid sorbent liquor 26. Absorber 11 may for example take the form of a tower which is packed with porous bodies so as to provide a high surface area for the gas-liquid contact. Other absorber apparatus as are known in the art can similarly be utilized. Pursuant to the invention, the sorbent liquor 26 comprises a preferably nonaqueous solvent having a high solubility for sulfur, typically in the range of from about 0.05 to 2.5 g-moles of sulfur per liter of solution. Sorbent liquor 26 as provided to absorber 11 includes sulfur dissolved in the nonaqueous solvent in the range of from about 0.05 to 2.5 g-moles of sulfur per liter of solution, together with a base (such as the aforementioned tertiary amines) having sufficient strength and sufficient concentration in respect to that of the hydrogen sulfide and sulfur to drive a reaction between the sulfur and hydrogen sulfide which results in formation of one or more nonvolatile polysulfides which are soluble in the solvent. In order to provide sufficient residence time for the reactions forming the polysulfide, a reactor vessel 15 is preferably provided downstream of the absorber. This vessel can also be physically present in a delay section at the base of the absorber tower. The reactor vessel can be of conventional construction such as a plug flow reactor. Total residence time for the reaction, whether carried out in the absorber alone, in the absorber and the reactor, or in the reactor alone, can be in the range of 5 to 30 minutes, with 15 minutes or so being typical. The polysulfide remains in solution in the solvent, and the spent sorbing liquor including the dissolved polysulfide is conveyed via line 13 to a regenerator 10. Since it is possible for certain polysulfide intermediates to separate as their concentration increases during practice of the invention (e.g., an amine-polysulfide "red oil" where the aforementioned base is a tertiary amine), a polysulfide solubilizing agent is preferably also present in sorbing liquor 26. Benzyl alcohol is a typical such solubilizing agent; however other agents such as benzhydrol, glycol, and mixtures of these several agents are suitable; and in addition the solubilizing function can be accomplished in some instances by one of the other components of the sorbent, such as the nonaqueous solvent or the base.

DOCUMENT-IDENTIFIER: US 6627377 B1

**\*\* See image for Certificate of Correction \*\***

TITLE: Positive photosensitive polyimide composition

Brief Summary Text (31):

As the preferred photosensitive aromatic diamines, firstly, dialkyl-diamino-bisphenyl sulfone and dialkoxy-diamino-biphenyl sulfone such as 3,3'-dimethyl-4,4'-diamino-biphenylsulfone and 3,3'-dimethoxy-4,4'-diamino-biphenylsulfone are exemplified. The polyimides containing such biphenyl sulfones are linear polymers having high mechanical strengths and high moduli of elasticity, so that they are studied as highly elastic polyimide fibers, and also as gas separation membranes because they can be made into films. They can be used as fibers or films, and also as photosensitive films. As shown in the Examples below, these polyimides containing biphenyl sulfone do not show photosensitivity even if Michler's ketone which is a sensitizer or a radical generator is added. It was discovered, however, that they are soluble in alkalis by irradiation with light after adding a quinone diazide compound. Even if the molecular weight (based on polystyrene) is changed to 30,000, 50,000 and 100,000, the polyimides are soluble in alkalis. From this fact, it is thought that the quinone diazide is photodecomposed to generate a radical and simultaneously to be converted to indene acid, and the product interacts with the polyimide groups and biphenyl sulfone groups, so that the polyimide is converted to be alkali-soluble. That is, by UV irradiation, the quinone diazide compound is photodecomposed and indene acid is further generated. As a result, the alkyl group or alkoxy group on the biphenyl group is activated so that the sulfone bond is cleaved, and indene acid is added thereto, thereby increasing the solubility of the polyimide in alkalis.

Brief Summary Text (32):

Additional preferred examples of the photosensitive aromatic diamines include 9,9-bis(aminophenyl)fluorene and 9,9-bis(aminoalkyl-phenyl)fluorene. The polyimides containing such fluorenes are linear polymers having high mechanical strengths and high moduli of elasticity, so that they are polyimides having excellent film properties, and having excellent properties when formed into gas separation membranes. They can be used as fibers or films, and also as photosensitive films. As shown in Examples below, these polyimides do not show photosensitivity even if Michler's ketone which is a sensitizer or a radical generator is added. It was discovered, however, they are converted to be soluble in alkalis by irradiation with light after adding a quinone diazide compound. Even if the molecular weight (based on polystyrene) is changed to 30,000, 50,000 and 100,000, they interact with the radical and the acid produced by photolysis of the quinone diazide to form alkali-soluble polyimides, which give clear positive-type images. More particularly, 9,9-bis(aminophenyl)fluorene is synthesized from fluorenone and aniline in the presence of an acid catalyst (Beilstein 13,III,548a). Fluorenone is a photosensitizer which is used as widely as Michler's ketone and benzanthrone. Although fluorenone-containing polyimides are sensitized by irradiation with light, they are usually not photodecomposed. It was discovered, however, if a quinone diazide co-exists, the quinone diazide generates a radical by irradiation with light, and radical becomes indene acid that interacts with the polyimide, so that the bis(aminophenyl)fluorene-containing polyimides are soluble in alkalis. This is presumably because that the SP<sup>3</sup> carbon structure at the 9-position of the bis(aminophenyl)fluorene group in the polyimide chain is temporarily stabilized by resonance and is changed to SP<sup>2</sup> carbon structure, so that the aniline group is eliminated and the polyimide chain is cleaved. Various fluorenone derivatives are known. For example, there are 2-nitro compounds, 2,7-dinitro compounds and 7-chloro compounds. Similarly, as for aniline, various derivatives such as 2-methylaniline and 2-methoxyaniline are known. From the above-described fluorenone derivatives and the aniline derivatives, various 9,9-bis(aminophenyl)fluorene derivatives are produced in the presence of an acid catalyst. These derivatives also constitute positive-type photosensitive compositions. By using benzanthrone compounds in place of the fluorenone, positive-type photosensitive polyimide compositions are also obtained.

First Hit

L13: Entry 199 of 261

File: USOC

Jul 6, 1965

DOCUMENT-IDENTIFIER: US 3193404 A

TITLE: Associated dye salts and method of forming colored indicia therewith

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3,198,404 at 160-290° C., resembling sodium azide in this respect. No explosion has ever been obtained under normal working conditions with this compound, which is very soluble in toluene. The only known member of this type, that from 5 Michler's hydrol and benzenesulfinic acid (called phenyl- (4,4'-bis(dimethylamino)-benzhydryl)-sulfone) is completely insoluble at room temperature in the usual solvents used in recording systems. It has been found by the author of the present invention, however, that the use of aliphatic sulfinic acids and the use of substituted aromatic sulfinic acids, where the substituent is one of a series of oil-solubility promoting (lipophilic) groups such as alkyl, halogen, ether, etc., gives solvent-soluble sulfinates which are stable, substantially colorless, and non-staining to skin, paper, and textile fibers. From the viewpoint of classic theory, these salts are for the most part the salts of weak bases and moderately strong acids. It is axiomatic in chemistry that the salts of strong bases-strong acids (sodium chloride, Crystal, 20 Violet Chloride, etc.) are always one hundred percent dissociated; but the salts of weak acids-strong bases, and of weak bases-strong acids may be more or less associated depending upon environmental conditions. It would appear that one method of adapting other dye bases for use in the present invention would be to lower their basic strength by suitable substitution. This is achieved in quanticule-donating systems by incorporating quanticule-attracting groups (nitro, trifluoromethyl, etc.) into the aryl group. It was found that Crystal Violet dye, for example, could be nitrated to give 2,2'-dinitro-4,4,4'-tris(dimethylamino)-triphenylcarbinol, a weak base. This weak base can form undissociated dye salts with suitable anions as well as dissociated dye salts with other anions. Similarly, 3,5-dinitro-4,4'-bis(dimethylamino)-triphenylcarbinol is easily prepared for use in the present invention as are other nitrated triarylmethane dye derivatives. While these nitrated dye bases containing only one nitro group are not always completely nonstaining, their associated salts are nonstaining and one can use associated salts for purposes where the free color bases are unsatisfactory. Although the associated salts of nitrated triarylmethane dye bases are not completely colorless, being a light orange in color, transfer sheets containing these salts are the same light yellow color as standard yellow commercial papers and may be substituted into any form where a pale-colored base web is not objectionable. The nonstaining nature of solutions of these salts renders their use far more attractive than the violet-colored, strongly-staining solutions of Methyl Violet Oleate. The use of other meta-directing groups in the nitro group to reduce the base strength of triarylmethane dyes, such as trifluoromethyl and N,N-dialkylsulfonamides, will also furnish intermediates for the dye salts of the present invention. In these cases, the parent carbinol bases are substantially colorless as are the resultant salts. The salts of the present invention are all water-insoluble. Inasmuch as water is ionizing in nature, colorless solutions of these associated salts in water, alcohol, etc. become colored upon addition of water; for this reason the Associated salts of the present invention should be used only in systems which do not have an appreciable water content. Water-soluble salts of triarylmethane color bases are discussed in my copending application, Stable 65 Triarylmethanesulfonic Acid Derivatives and Method of Forming Colored Indicia

Therewith, Serial No. 200,056, filed June 5, 1962. The author has found that certain unfired silicates such as diatomaceous earth, kaolin, and bentonite possess high ionizing properties apart from their acid-base and oxidation-reduction properties. A clay molecule may be pictured as a large molecule of polymerized silica containing calcium, iron, and other cations. The iron silicate structure gives oxidizing properties to the clay and the calcium silicate gives acidic and ion-exchange properties to the clay, but it is the polymerized silica structure which gives a high electric dipole moment over the surface of the clay; - This electric dipole is stable until the clay is calcined at high temperature, at which point the electric dipole moment disappears; and the aforementioned clays lose their ionizing properties. Although other workers in the recording field, notably Bjorksten, Green, and Bour, have utilized certain silicates in recorders, no worker has yet utilized the clays as other than chemical reagents. It is the purpose of this application to describe a practical utilization of the ionizing properties of silicates possessed of a high dipole moment. The compounds of the present invention offer certain advantages over the color bases from which they are derived: (1) As the method of color formation by dissociation is different from the method of color formation by reaction of a dye base with an acid, there is oftentimes a marked increase in the rate of reaction. Dinitro Crystal Violet Base, for example, when pure forms a colored salt with acid silicates only very slowly (3-5 minutes or longer); but the dissociation of Dinitro Crystal Violet Base to the colored ion under the influence of the silicate's electric dipole moment is immediate, and an intense colored print appears immediately upon recording with this azide. (2) The stability of certain color bases, such as mononitro Crystal Violet Base, which have logarithmic dissociation constants between 5 and 7, is improved, and this is of value in manifolding - Records which must be stored indefinitely before use. (3) The associated salts formed can be more safely handled without chemical staining than can the more reactive color bases because of lower water-solubility (i.e., they are less soluble in the skin perspiration). While the compounds of the present invention cannot be used in aqueous solutions and are more reactive than the water-soluble triarylmethane-sulfonates, which are disclosed in my copending application, they do, however, have applications in recording systems where a nonvolatile color-former is desired which will record colored on selected areas from a lipophilic solvent. Description Alichler's hydrol (4,4' - bis-(dimethylamino)-benzhydrol), ethyl hydrol (4,4'-bis-(diethylamino)-benzhydrol), Dinitro Crystal Violet Carbinol and other compounds of this type are dissolved in glacial acetic acid to form the intensely colored acetate. To this colored dye solution is added the desired sulfonic acid either as the free sulfonic acid or as its sodium salt (the sodium salt form is preferred because the sulfonic acid salts have greater storage stability than the free sulfonic acids which tend to polymerize upon standing in the air) until the intense color is discharged or until the solution becomes a markedly lighter color. The sulfinate may deposit at this time or may be retained in solution. The solution is then poured into cold water or cold ammonium hydroxide solution to precipitate the water-insoluble sulfinate. The precipitate is collected, washed with water, dried, and recrystallized from alcohol to give the colorless sulfinate. The reaction may be carried out in dilute aqueous acids or in acidic alcohol. All that is necessary is that the dye base should be converted to the colored salt before adding the sulfinate ion. The sulfonic acids may be obtained by decomposition of a sulfonyl-dioxide containing diazonium salt solution (Gatfermann reaction), by reduction of the sulfonyl chloride with zinc dust or sodium sulfite, or by the aluminum chloride catalyzed addition of sulfur dioxide to an olefinic or aromatic compound. The yield of colorless sulfinate is usually a very high percentage and is often quantitative. The melting points of these colorless dye salts depends on the rate of heating and cannot be used to characterize

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TITLE: Fluid based analysis of multiple analytes by a sensor array

Detail Description Paragraph:

[0061] FIGS. 4A-F depict a sequence of processing steps for the formation of a cavity and a planar top diaphragm Fabry-Perot sensor on the bottom surface of a silicon based supporting member. A sacrificial barrier layer 262a/b is deposited upon both sides of a silicon supporting member 260. The silicon supporting member 260 may be a double-side polished silicon wafer having a thickness ranging from about 100 .mu.m to about 500 .mu.m, preferably from about 200 .mu.m to about 400 .mu.m, and more preferably of about 300 .mu.m. The barrier layer 262a/b may be composed of silicon dioxide, silicon nitride, or silicon oxynitride. In one embodiment, the barrier layer 262a/b is composed of a stack of dielectric materials. As depicted in FIG. 4A, the barrier layer 262a/b is composed of a stack of dielectric materials which includes a silicon nitride layer 271a/b and a silicon dioxide layer 272a/b. Both layers may be deposited using a low pressure chemical vapor deposition ("LPCVD") process. Silicon nitride may be deposited using an LPCVD reactor by reaction of ammonia (NH.sub.3) and dichlorosilane (SiCl.sub.2H.sub.2) at a gas flow rate of about 3.5:1, a temperature of about 800.degree. C., and a pressure of about 220 mTorr. The silicon nitride layer 271a/b is deposited to a thickness in the range from about 100 .ANG. to about 500 .ANG., preferably from 200 .ANG. to about 400 .ANG., and more preferably of about 300 .ANG.. Silicon dioxide is may be deposited using an LPCVD reactor by reaction of silane (SiH.sub.4) and oxygen (O2) at a gas flow rate of about 3:4, a temperature of about 450.degree. C., and a pressure of about 110 mTorr. The silicon dioxide layer 272a/b is deposited to a thickness in the range from about 3000 .ANG. to about 7000 .ANG., preferably from 4000 .ANG. to about 6000 .ANG., and more preferably of about 5000 .ANG.. The front face silicon dioxide layer 272a, in one embodiment, acts as the main barrier layer. The underlying silicon nitride layer 271a acts as an intermediate barrier layer to inhibit overetching of the main barrier layer during subsequent KOH wet anisotropic etching steps.

Detail Description Paragraph:

[0075] A naturally occurring or synthetic receptor may be bound to a polymeric resin in order to create the particle. The polymeric resin may be made from a variety of polymers including, but not limited to, agarous, dextrose, acrylamide, control pore glass beads, polystyrene-polyethylene glycol resin, polystyrene-divinyl benzene resin, formylpolystyrene resin, trityl-polystyrene resin, acetyl polystyrene resin, chloroacetyl polystyrene resin, aminomethyl polystyrene-divinylbenzene resin, carboxypolystyrene resin, chloromethylated polystyrene-divinylbenzene resin, hydroxymethyl polystyrene-divinylbenzene resin, 2-chlorotrityl chloride polystyrene resin, 4-benzyloxy-2'4'-dimethoxybenzhydrol resin (Rink Acid resin), triphenyl methanol polystyrene resin, diphenylmethanol resin, benzhydrol resin, succinimidyl carbonate resin, p-nitrophenyl carbonate resin, imidazole carbonate resin, polyacrylamide resin, 4-sulfamylbenzoyl-4'-met- hylbenzhydramine-resin (Safety-catch resin), 2-amino-2-(2'-nitrophenyl)p- ropionic acid-aminomethyl resin (ANP Resin), p-benzyloxybenzyl alcohol-divinylbenzene resin (Wang resin), p-methylbenzhydramine-diviny- lbenzene resin (MBHA resin), Fmoc-2,4-dimethoxy-4'-(carboxymethyloxy)-benz- hydramine linked to resin (Knorr resin), 4-(2',4'-Dimethoxyphenyl-Fmoc-a- minomethyl)-phenoxy resin (Rink resin), 4-hydroxymethyl-benzoyl-4'-methylb- enzhydramine resin (HMBA-MBHA Resin), p-nitrobenzophenone oxime resin (Kaiser oxime resin), and amino-2,4-dimethoxy-4'-(carboxymethyloxy)-benzh- ydramine handle linked to 2-chlorotrityl resin (Knorr-2-chlorotrityl resin). In one embodiment, the material used to form the polymeric resin is compatible with the solvent in which the analyte is dissolved. For example, polystyrene-divinyl benzene resin will swell within non-polar solvents, but does not significantly swell

within polar solvents. Thus, polystyrene-divinyl benzene resin may be used for the analysis of analytes within non-polar solvents. Alternatively, polystyrene-polyethylene glycol resin will swell with polar solvents such as water. Polystyrene-polyethylene glycol resin may be useful for the analysis of aqueous fluids.